

Aluminum, atomic emission spectrometric, d-c plasma

Parameters and Codes:

Aluminum, dissolved, I-1054-85 ($\mu\text{g/L}$ as Al): 01106

Aluminum, total recoverable, I-3054-85 ($\mu\text{g/L}$ as Al): 01105

Aluminum, suspended recoverable, I-7054-85 ($\mu\text{g/L}$ as Al): 01107

1. Application

1.1 This method may be used to analyze finished water, natural water, industrial water, and water-suspended sediment containing from 10 to 1000 $\mu\text{g/L}$ of aluminum. Samples containing more than 1000 $\mu\text{g/L}$ aluminum and (or) with specific conductances greater than 10,000 $\mu\text{S/cm}$ need to be diluted.

1.2 Suspended recoverable aluminum is calculated by subtracting dissolved aluminum from total recoverable aluminum.

1.3 Total recoverable aluminum in water-suspended sediment needs to undergo a preliminary digestion-solubilization by method I-3485 before being determined.

2. Summary of method

Aluminum is determined by a direct-reading emission spectrometer which utilizes a d-c argon plasma as an excitation source (Johnson and others, 1979 a,b, 1980). A mixture of lithium chloride, sulfuric acid, and glycerin is added to samples and standards to provide a common background matrix and to compensate for viscosity changes. The liquid mixture is then converted by a ceramic nebulizer into a fine aerosol and introduced into the plasma via a plastic spray chamber and Pyrex injection tube. Aluminum is determined on the basis of the average of two replicate exposures, each of which is performed on a 10-second integrated intensity. Calibration is performed by standardization with a high-standard solution and a blank.

3. Interferences

Stray-light effects in a high-resolution, single-element d-c argon plasma emission spectrometer are negligible.

4. Apparatus

4.1 Spectrometer, Spectrometrics, Spectrospan IV with d-c argon plasma or equivalent, with Echelle optics, printer, autosampler, and peristaltic pump.

4.2 Refer to manufacturer's manual to optimize instrument for the following:

Plasma viewing position +1 (fig. 1)

Gas ----- Argon

Sleeve pressure ----- 50 psi

Nebulizer pressure --- 25 psi

Entrance slit ----- 25X 300 μm

Exit slit ----- 50X 300 μm

Voltage ----- 1000 V

Wavelength ----- 308.215 nm

Signal amplification-- 40- to 60-per-
cent full-scale
(1000 $\mu\text{g/L}$)

5. Reagents

5.1 *Aluminum standard solution I*, 1 mL= 100 μg Al: Dissolve 0.100 g aluminum powder in a minimum of 6M HCl using a Teflon beaker. Heat to increase rate of dissolution. Add 10.0 mL 6M HCl and dilute to 1,000 mL with demineralized water. Store in plastic bottle.

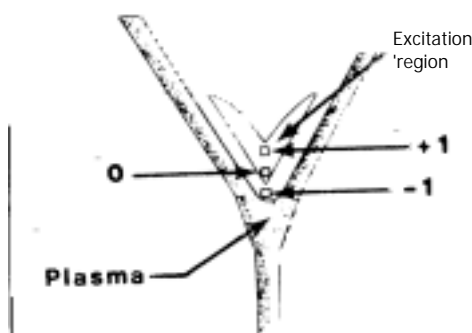


Figure 1. - Plasma position on entrance slit for aluminum

5.2 *Aluminum standard solution II*, 1.00 mL=10.0 µg Al: Dilute 100.0 mL aluminum standard solution I to 1000 mL with demineralized water. Store in plastic bottle.

5.3 *Aluminum working standard*, 1.00 mL=1.00 µg Al: Dilute 100.0 mL aluminum standard solution II to 1000 mL with demineralized water. Store in plastic bottle.

5.4 *Glycerin*, USP.

5.5 *Hydrochloric acid*, concentrated (sp gr 1.19), Ultrex or equivalent.

5.6 *Hydrochloric acid*, 6M Add 500 mL concentrated HCl (sp gr 1.19) to 400 mL demineralized water and dilute to 1 L with demineralized water.

5.7 *Lithium chloride*, LiCl, reagent-grade.

5.8 *Matrix modifier*. Dissolve 367 g LiCl in 1,000 mL demineralized water. Allow the solution to cool. Transfer to a 4-L polyethylene container, and add with stirring 2,000 mL of glycerin. In a Teflon beaker slowly add with stirring 400 mL concentrated H₂SO₄ to 400 mL demineralized water. When the dilute acid has reached room temperature, add the acid slowly, with stirring, to the glycerin-LiCl mixture Dilute to 4,000 mL with demineralized water.

5.9 *Sulfuric acid*, concentrated (sp gr 1.84), Ultrex or equivalent.

6. Procedure

6.1 Pipet 10.0 mL sample into a disposable plastic test tube.

6.2 Pipet 100 mL demineralized-water blank and working standard into plastic bottles.

6.3 Add 2.0 mL matrix modifier to the sample and 20.0 mL to the blank and working standard.

6.4 Place plastic caps on the tube and bottles and mix well.

6.5 Refer to manufacturer's manual for computer-operating and wavelength-optimization procedures. Use the prepared blank and aluminum working standard for instrument calibration and all subsequent recalibrations.

6.6 Refer to manufacturer's manual for auto-sampler-operating procedures. Pour samples in autosampler tray, positioning a blank and working standard after every 3 samples for recalibration. Begin analysis (NOTE 1).

NOTE 1. Because of thermal instability inherent with the high-resolution spectrometer, repeat the

analytical line if the aluminum standard drifts more than 3 percent.

7. Calculations

The computer system is designed so that the blank and the 1,000 µg/L of aluminum standard are used to establish a two-point calibration curve. The system will convert instrument intensity readings to analytical concentrations. The printer display includes the blank and working-standard instrument intensity readings, blank and standard concentrations, sample instrument intensity readings, sample concentrations, average of sample concentrations, and standard deviation.

8. Report

Report aluminum, dissolved (01106), total-recoverable (01105), and suspended-recoverable (01107), concentrations as follows: less than 100 µg/L, nearest 10 µg/L; 100 µg/L and above, two significant figures.

9. Precision

9.1 Precision, based on 14 to 18 determinations by a single operator during a 47-day period, expressed in terms of standard deviation and percent relative standard deviation, is as follows:

Number of determination	Mean (µg/L)	Standard deviation (µg/L)	Relative Standard deviation (percent)
17	12.9	1.9	14.7
14	30.5	3.0	9.8
14	73.1	8.9	12.2
15	132	11	8.3
18	221	6.0	2.7
18	437	18	4.1
18	763	32	4.2

9.2 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable aluminum will be greater than that reported for dissolved aluminum.

References

- Johnson, G.W, Taylor, H. E., and Skogerboe, R.K, 1979a, Determination of trace elements in natural waters by the D.C. argon-plasma, multielement atomic emission spectrometer (DCP-MAES) technique: Spectrochimica Acta, v. 34B, p. 197-212.
- _____, 1979b, Evaluation of spectral interferences associated with a direct current plasma-multielement atomic emission spectrometer (DCP-MAES) system: Applied Spec-

troscopy, v. 33, p. 451-456.

____1980, Characterization of an interelement enhancement effect in a dc plasma atomic emission spectrometry system: Applied Spectroscopy, v. 34, p. 19-24.